

CHANGE NOTICES ARE NOT
CUMULATIVE AND SHALL BE
RETAINED UNTIL SUCH TIME
AS THE ENTIRE STANDARD
IS REVISED

Fed. Test Method Std. No. 191
December 31, 1968
Change Notice 1
July 10, 1970

FEDERAL STANDARD

TEXTILE TEST METHODS

The following changes to Fed. Test Method Std. No. 191 dated December 31, 1968 have been approved by the Commissioner, Federal Supply Service, General Services Administration for the use of all Federal agencies.

1. CHANGES

1.1 Section 6 - Numerical Index updated.

1.2 Section 7 - Alphabetical Index updated.

1.3 Section 10 - Supersession Data, Source Information, and Interested Agencies updated.

1.4 TM 2011.1 - Added a spectrophotometer, a blank which is required for the initial adjustment of the spectrophotometer. The use of a buffer solution to preclude the influence of interfering ions. A standard curve limited to 20 to 60 microgram range. That the analysis be based on the dry weight of the specimen.

1.5 TM 3810.1 Deleted the requirement for the use of a rotary blade food cutter. Format revised.

1.6 TM 4010.1 Format revised.

1.7 TM 4021.1 Added conversion factors for expressing linear density.

1.8 TM 4050.1 Format revised.

1.9 TM 4052.1 Format revised.

1.10 TM 4054.1 Format revised.

1.11 TM 4100.1 Deleted the constant-rate-of load apparatus. Added new paragraph for determining tenacity. Added specific description of clamps used for holding specimen. Format revised.

1.12 TM 4104.1 Format revised.

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PAGE OF THIS STANDARD

FSC 8300

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1.13 TM 4108.1 Added specific description of apparatus and drawing of split-drum type clamp. Format revised.

1.14 TM 4110.1 Format revised.

1.15 TM 4112.1 Format revised.

1.16 TM 4308.1 Source of supply updated. Format revised.

1.17 TM 5030.1 Sources of supply updated.

1.18 TM 5206.1 Source of supply updated.

1.19 TM 5308.1 Source of supply updated. Format revised.

1.20 TM 5450.1 Source of supply updated.

1.21 TM 5556.1 Table II revised.

1.22 TM 5903.1 Sources of supply updated.

1.23 TM 5920.1 Format revised.

1.24 TM 7308.1 Source of supply updated. Format revised.

2. ADDITIONS

2.1 The following methods have been added to this Standard:

2.1.1 TM 2013. Fluorine Content of Textile Materials.

2.1.2 TM 2015. Sodium Salt of 5-Chloro-2- [4 Chloro-2- [3-(3, 4 Dichlorophenyl) -Uredio] -Phenoxy] Benzenesulfonate Content.

2.1.3 TM 5309. Abrasion Resistance of Textile Webbing.

2.1.4 TM 5764. Insect Resistance of Textile Materials.

3. DELETIONS

3.1 The following methods have been deleted from this Standard:

3.1.1 TM 1533. Identification by Softening Point of Thermoplastic Fibers. The test requirements are contained in TM 1534, Melting Point of Synthetic Fibers.

3.1.2 TM 4020. Yarn Number; Cotton-Yarn Method. The test requirements are contained in TM 4021, Yarn Number (Linear Density) of Yarn from Package.

3.1.3 TM 5910. Burning Rate of Cloth; 30° Angle.

SECTION 6

NUMERICAL INDEX OF TEST METHODS

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1110	Identification of Silk
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1210	Identification of Flax
1220	Identification of Hemp
1230	Identification of Ramie
1240	Identification of Manila (Abaca'); Microscopic Method
1241	Identification of Manila (Abaca'); Ash Method
1250	Identification of Sisal; Microscopic Method
1251	Identification of Sisal; Ash Method
1260	Identification of Jute
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1400	Identification of Asbestos
1410	Identification of Glass
1500	Identification of Rayon, Viscose
1510	Identification of Rayon, Acetate

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1530	Identification of Nylon
1534	Melting Point of Synthetic Fibers
1540	Identification of Vinylidene Chloride Fibers
1550	Identification of Vinyl Chloride-Acetate Copolymer Fibers
1600	Identification of Synthetic Fibers by Generic Class
1700	Identification of Dyes on Animal Fibers

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2011.1	Dihydroxydichlorodiphenyl Methane Content, Colorimetric Method
2012	Dihydroxydichlorodiphenyl Methane Content, Parr Chloride Method
2013	Fluorine Content of Textile Materials
2015	Sodium Salt of 5-Chloro -2- [4 Chloro -2- (3- (3,4 Dichloro-phenyl) -Ureido] -Phenoxy] Benzenesulfonate Content
2020	Presence of Labile Sulfur in Textile Materials
2050	Copper content of Textiles, Electrolytic Method
2051	Copper Content of Textiles, Polarographic Method
2053	Small Amounts of Copper and Manganese in Textiles
2060	Copper-8-Quinolinolate Content of Textiles, Spectrophotometric Method
2100	Wool Content, Acid Method
2101	Wool Content, Alkali Method
2102	Wool Content, Hypochlorite Method

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2110	Silk Content of Fiber Mixtures
2111	Silk Fiber Content of Silk Textiles (Especially Weighted Silk)
2510	Cellulose Acetate Content of Fiber Mixtures, Acetic Acid Method
2511	Cellulose Acetate Content of Fiber Mixtures, Acetone Method
2530	Nylon Content of Fiber Mixtures
2600	Moisture Content, Oven Method
2601	Moisture Content, Oven-Balance Method
2610	Nonfibrous Materials in Cotton, Acid Method
2611	Nonfibrous Materials in Cotton, Enzyme Method
2620	Nonfibrous Materials in Linen Textiles
2800	Wool Fiber Damage, Alkali Solubility Method
2810	Acidity (pH) of Textiles, Colorimetric Method
2811	pH of Textiles, Electrometric

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3810.1	Becker Value of Cordage Fiber
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Yarn, Thread, Cordage

4010.1	Length-Weight Relation; Thread; Yards Per Pound
4021.1	Yarn Number (Linear Density) of Yarn from Package

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4052.1	Twist in Single Yarns; Turns Per Inch
4054.1	Twist and Twist Contraction; Ply Yarns
4100.1	Breaking Strength, Elongation, Tenacity; Thread Yarn; Single Strand
4102	Strength and Elongation, Breaking; Small Cords; Single Strand
4104.1	Breaking Strength; Thread and Yarn; Skein Method
4106	Strength, Breaking; Heavy Cordage (Rope)
4108.1	Breaking Strength and Elongation; Textile Webbing, Tape and Braided Items
4110.1	Crimp in Yarns from Cloth; Dead-Load Method
4112.1	Crimp in Yarns from Cloth; Load-Elongation Method
4308.1	Abrasion Resistance of Yarn, Thread, and Light Cordage; Uniform-Abrasion (Schiefer) Method
4500	Water Absorption, Dynamic; Tumble Jar Method
4502	Water Absorption; Thread, Cord, Braid, Tape, Webbing; Immersion Method
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4800	Weathering Resistance; Yarn, Cordage; Natural Weathering Method
4804	Weathering Resistance; Yarn, Thread, Cordage; Accelerated Weathering Method
4830	Leaching Resistance; Cordage; Standard Method
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5041	Determination of Weight of Textile Materials: Small Specimen Method
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5302	Abrasion Resistance of Cloth; Inflated Diaphragm (Stoll) Method
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5306	Abrasion Resistance of Cloth; Rotary Platform, Double-Head (Taber) Method
5308.1	Abrasion Resistance of Cloth; Uniform Abrasion (Schiefer) Method
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5510	Dry Cleaning Resistance of Cloth With Water-Resistant Finish; Rotating Wheel Method
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- 5622 Colorfastness to Wet Cleaning of Textile Materials; (Associated with Dry Cleaning)
- 5630 Colorfastness of Textile Materials to Water
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6011	Water Absorption; Cordage
4500	Water Absorption, Dynamic; Tumble Jar Method
4502	Water Absorption; Thread, Cord, Braid, Tape, Webbing; Immersion Method
5520	Water Resistance of Cloth; Drop Penetration Method
5500	Water Resistance of Cloth; Dynamic Absorption Method
5502	Water Resistance of Cloth; Immersion Absorption Method
5514	Water Resistance of Cloth; Low Range Hydrostatic Pressure Method
5524	Water Resistance of Cloth; Rain Penetration Method
5522	Water Resistance of Cloth; Water Impact Penetration Method
5516	Water Resistance of Cloth Water Permeability, Hydrostatic Pressure Method
5526	Water Resistance of Cloth with Hydrophobic Finish; Spray Method
5512	Water Resistance of Coated Cloth; High Range, Hydrostatic Pressure Method

Alphabetical Index (Cont'd)

5504	Water Resistance of Coated Cloth; Spray Absorption Method
5528	Water Resistance of Coated Cloth; Spray Method
4504	Water Resistance, Vertical Rise Wicking, Thread
5804	Weathering Resistance of Cloth; Accelerated Weathering Method
5800	Weathering Resistance of Cloth; Natural Weathering Method
4800	Weathering Resistance; Yarn, Cordage; Natural Weathering Method
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5040	Weight of Cloth; Cut, Roll or Bolt Method
2100	Wool Content, Acid Method
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2102	Wool Content, Hypochlorite Method
2800	Wool Fiber Damage, Alkali Solubility Method
4021.1	Yarn Number (Linear Density) of Yarn From Package
5050	Yarns Per Inch in Woven Cloth

DIHYDROXYDICHLORODIPHENYL METHANE CONTENT,
COLORIMETRIC METHOD

1. SCOPE

1.1 This method is intended for determining the dihydroxydichlorodiphenyl methane (2, 2' methylene bis - 4 - chlorophenol) content of textile materials that have been heated with this compound to prevent the formation of mildew.

2. TEST SPECIMEN

2.1 The specimen shall be one gram composite of the material prepared as described in 5.1.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS

4.1 Apparatus

4.1.1 Spectrophotometer and accessories.

4.1.2 Volumetric flasks.

4.1.3 Microburette.

4.1.4 Beakers.

4.1.5 Pipettes.

4.1.6 Air oven.

METHOD 2011.1

4.1.7 Weighing bottle.

4.2 Reagents.

4.2.1 Crystallized 4-aminoantipyrine solution, freshly prepared. The solution shall be prepared by dissolving 2 ± 0.001 grams of 4-aminoantipyrine, m.p. 108° - 109° C. in 100 cc of distilled water.

4.2.2 Potassium ferricyanide solution, freshly prepared. The solution shall be prepared by dissolving 8 ± 0.001 grams of reagent grade potassium ferricyanide in 100 cc of distilled water.

4.2.3 Sodium carbonate solution, approximately 0.03 percent, pH between 10.4 and 10.6. Distilled water shall be used. The pH should be rechecked just prior to using.

4.2.4 Dihydroxydichlorodiphenyl methane(2, 2' methylene bis-4-chlorophenol) "G-4 Technical" or equal (see 7.1).

4.2.5 Buffer solution (distilled water), 2.47 percent boric acid, 0.4 percent sodium hydroxide, pH of 9.1 to 9.2 (adjust with additional sodium hydroxide, if necessary.)

5. PROCEDURE

5.1 Preparation of specimen. Three specimens not less than two grams each shall be cut from the sample unit. One specimen shall be cut from each edge of the sample unit but will not include the selvage except for narrow fabrics such as braid, tape or webbing when it is not practical or possible. The third specimen shall be taken from the middle of the sample unit. No two specimens shall contain the same warp or filling yarns except narrow fabrics such as braid, tape or webbing when it is not practical or possible.

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5.1.1 The three specimens taken from the sample unit shall be cut in small pieces approximately $1/8$ inch (3mm) square and thoroughly mixed to form a composite sample.

FLUORINE CONTENT OF TEXTILE MATERIALS

1. SCOPE

1.1 It is intended that this method be used to determine the fluorine content of materials which have fluorine compounds for moth repellency.

2. TEST SPECIMEN

2.1 The test specimen shall be 0.5 gram composite of the material prepared as described in 5.1.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, two specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Fluorine determination apparatus having a 300 cc flask with a no. 40/50 joint (see 7.1).

4.1.2 Burette

4.1.3 Beakers.

4.1.4 200 cc volumetric flask.

4.1.5 Bunsen burner or electric heater.

4.2 Reagents.

4.2.1 50 percent sulfuric acid (H_2SO_4) by volume in distilled water.

4.2.2 0.01N thorium nitrate $Th(NO_3)_4$ standardized against a standard sodium fluoride (NaF) solution.

4.2.3 0.1N hydrochloric acid (HCl) solution.

4.2.4 10 percent sodium hydroxide (NaOH) solution.

4.2.5 Indicator: 0.05 percent aqueous solution sodium alizarin sulfonate.

5. PROCEDURE

5.1 Preparation of specimen. Three specimens not less than two grams each shall be cut from the sample unit. One specimen shall be cut from each edge of the sample unit but will not include the selrage. The third specimen shall be taken from the center of the sample unit. No two specimens shall contain the same warp or filling yarns.

5.1.1 The three specimens taken from the sample unit shall be cut in small pieces approximately 1/8 inch(3mm) square and thoroughly mixed to form a composite sample.

5.2 Transfer specimen to the 300 cc flask of the fluorine apparatus (see figure 2013) and add 40 cc of 50 percent sulfuric acid. Connect the flask to the fluorine apparatus. Immerse the lower end of condenser in 20 cc of distilled water in the receiving beaker to insure the solution of gases.

5.3 Heat gradually to dissolve specimen and then start distillations with a high heat (Bunsen burner or electric heater). Avoid localized overheating when using Bunsen burner by protecting the flask with asbestos sheeting having a relatively small hole in the asbestos sheet placed directly under the bottom of the flask.

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5.4 Maintain liquor temperature between 136° and 140° C by dropping distilled water from a separatory funnel inserted in the neck of the flask. Collect 200 cc but not less than 176 cc of distillate. Transfer distillate to 200 cc volumetric flask; if less than 200 cc is distilled make up to volume with distilled water. Shake well, then allow any fats or waxes that may have been distilled over to rise in the neck of the flask. Remove any of the fats or waxes that are present and again make up volume with distilled water.

5.5 Titrate two 20 cc aliquots of the distillate. To each aliquot add two drops sodium alizarin sulfonate indicator and one drop of

5.3 Preparation of standard curve. 0.2 of a gram of dihydroxydichlorodiphenyl methane shall be weighed to the nearest milligram, dissolved in a few cc of acetone, transferred to a 200 cc volumetric flask, and filled to the mark with acetone. One cc of this solution shall be pipetted into a 100 cc volumetric flask and diluted to the mark with the 0.03 percent sodium carbonate solution. This solution which contains 10 micrograms of dihydroxydichlorodiphenyl methane per cc shall be used for suitable aliquots covering the range of 20 to 60 micrograms. (2 cc equals 20 micrograms, etc.). Each aliquot shall be placed in a 25 cc volumetric flask. The color shall be developed and the percent transmission determined as in 5.2.1. The standard curve shall be plotted on linear graph paper, plotting percent transmission versus micrograms of dihydroxydichlorodiphenyl methane in 25 cc of solution. The standard curve shall be drawn by connecting consecutive points between 20 and 60 micrograms by straight lines.

5.4 Calculations.

5.4.1 Unless otherwise specified, the dihydroxydichlorodiphenyl methane content of the specimen shall be calculated as follows:

$$\text{dihydroxydichlorodiphenyl methane, percent} = \frac{0.02A}{B \times S}$$

Where: A = Dihydroxydichlorodiphenyl methane concentration from the standard curve in micrograms.

B = Aliquot of 200 cc test solution taken, cubic centimeters.

S = Weight of oven dried specimen, grams.

6. REPORT

6.1 The dihydroxydichlorodiphenyl methane content of the sample unit shall be the average of the results obtained from the specimens tested and shall be reported to the nearest 0.1 percent.

6.1.1 The individual values for each individual specimen used to calculate the average shall also be reported.

7. NOTES

7.1 Samples of "G-4 Technical" can be obtained from Sindar Corp., a Division of Givaudan Corporation, 125 Delawanna Avenue, Clifton, New Jersey 07014.

10 percent sodium hydroxide. The addition of the sodium hydroxide will produce a light violet color (see 7.2, 7.3). Titrate the distillate with 0.1N hydrochloric acid to a yellow coloration then add five drops in excess (see 7.4). Titrate the resultant solution to a faint but definite pink coloration with 0.01N thorium nitrate solution.

5.6 The percent fluorine shall be calculated as follows:

Percent fluorine = cubic centimeters x 0.38

Where: cubic centimeters = cc of 0.01N $\text{Th}(\text{NO}_3)_4$ required for 20 cc aliquot titration. For 0.5g. specimen

5.6.1 In cases where an alternate calculation formula is necessary because the weight is less or more than 0.5 g, the percent fluorine (F) shall be calculated as follows:

$$\text{Percent fluorine} = \frac{\text{cc of } \text{Th}(\text{NO}_3)_4 \times \text{N of } \text{Th}(\text{NO}_3)_4 \times 0.19 \times 100}{\text{Sample weight (g)}}$$

6. REPORT

6.1 The percent fluorine shall be the average of specimens tested from a sample unit and shall be reported to the nearest 0.1 percent.

6.2 Individual results used to calculate the average shall also be reported.

7. NOTES

7.1 The apparatus for fluorine determination used in this method may be purchased from Ace Glass Inc., Vineland, N. J. 08360 as No. 6430.

7.2 Twenty cc aliquot of distillate should never require more than one drop of 10 percent sodium hydroxide solution. When more is required, contamination of the distillate by decomposition products is indicated. The distillate shall be discarded and a new specimen tested.

7.3 Sodium ion concentration in the distillate should be kept to a minimum since these ions cause erroneous (high) results.

7.4 The pH of the solution taken for titration is important. The pH provided by the addition of five drops excess 0.1N HCl is ideal (i.e. 3.2). The use of buffers is not recommended because the end point is not well defined even in the absence of a buffer.

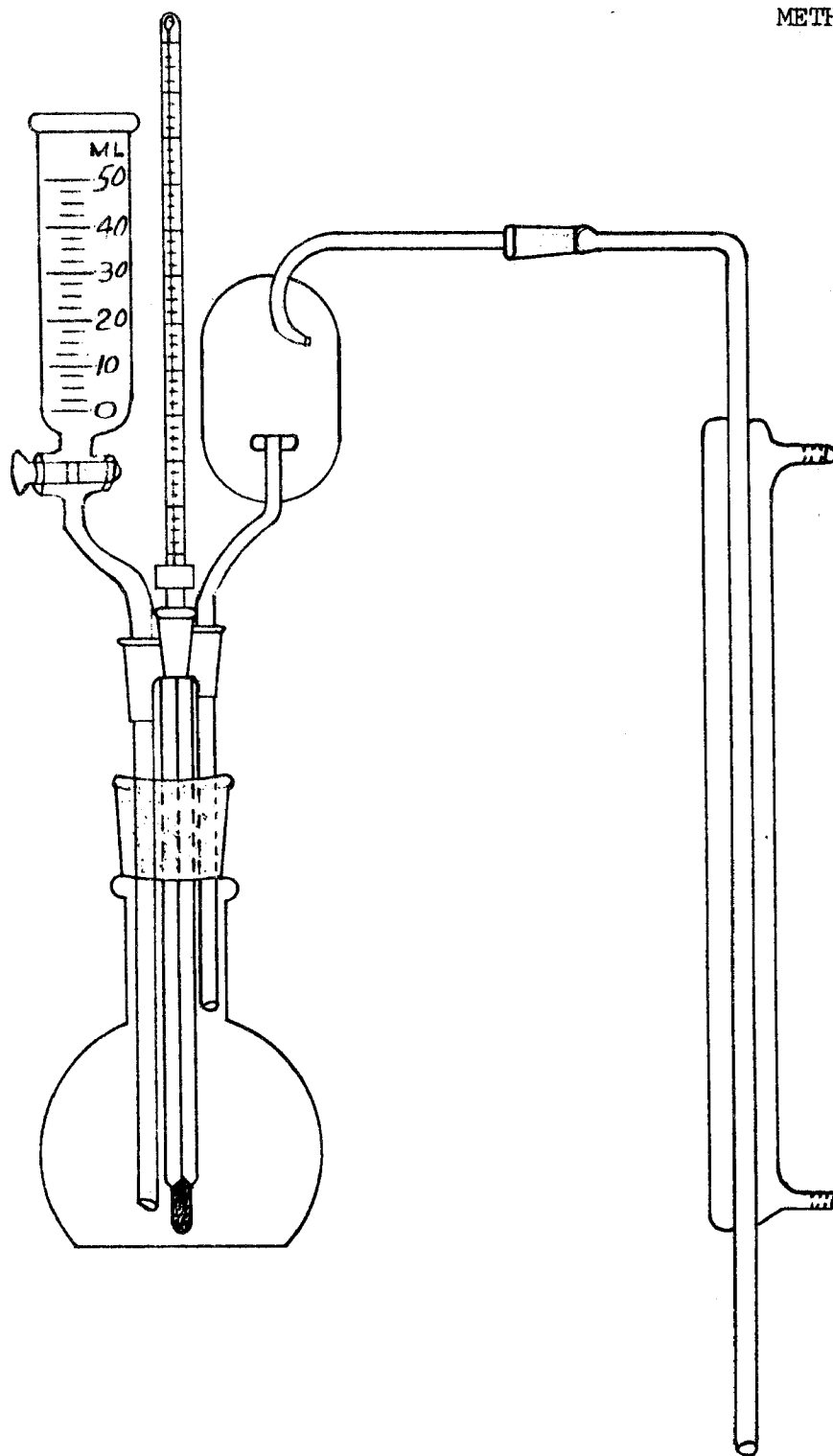


FIGURE 2013 - Test apparatus for fluorine content of textile materials.

SODIUM SALT OF 5-CHLORO-2-[4CHLORO-2-[3-(3, 4 DICHLOROPHENYL)-
UREIDO] -PHENOXY] BENZENESULFONATE CONTENT

1. SCOPE

1.1 This method is intended to determine the sodium-5-chloro-2-[4 chloro-2-[3-(3,4 dichlorophenyl)-ureido] -phenoxy] benzenesulfonate content of woolen textile materials that have been treated with this compound as a mothproofing agent (see 7.2).

2. TEST SPECIMEN

2.1 When the material to be tested is 100 percent wool, the specimen shall be 500 ± 50 milligrams.

2.2 When the material to be tested is a blend of polyester and wool, the specimen shall be 1000 ± 100 milligrams.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified, two specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS

4.1 Apparatus.

4.1.1 Electric heater with variable control.

4.1.2 250 cc, round bottom, single neck, alkali resistant, Pyrex glass flask.

4.1.3 250 cc trap bulb and connecting arm (see 7.1).

4.1.4 Graham condenser (jacket 300 mm, long).

4.1.5 Funnel.

METHOD 2015

- 4.1.6 500 cc volumetric flasks.
- 4.1.7 1000 cc volumetric flasks.
- 4.1.8 Pipettes.
- 4.1.9 Glass beads.
- 4.1.10 Spectrophotometer or filter photometer with a green filter having a maximum transmission at approximately 500 nanometers.
- 4.1.11 Silicone stopcock lubricant.
- 4.1.12 Potassium Iodide-Starch test paper.
- 4.1.13 Congo Red test paper.
- 4.1.14 Red and blue litmus paper.
- 4.1.15 Analytical balance.
- 4.1.16 Air oven.
- 4.2 Reagents.
- 4.2.1 2.0 N potassium hydroxide (KOH), 112 grams of potassium hydroxide pellets ACS, per 1000 cc of solution.
- 4.2.2 1.0 N hydrochloric acid (HCl), 85 cc of hydrochloric acid, 37.5 percent concentrated, ACS per 1000 cc of solution.
- 4.2.3 37.5 percent concentrated hydrochloric acid, ACS.
- 4.2.4 0.1 N sodium nitrite (NaNO_2), 6.9 grams sodium nitrite ACS, per 1000cc of solution. Sodium nitrite is subject to decomposition and should be made up fresh.
- 4.2.5 0.01 molar benzoyl-H-acid (1 naphthol 3, 6 disulfonic acid, 8 benzamido), 8.9 grams 50 percent benzoyl-H-acid per 1000 cc of solution. Store protected from light, (see 7.1).

4.2.6 1.0 N sodium bicarbonate (NaHCO_3), 42 grams sodium bicarbonate ACS, per 1000 cc of solution.

4.2.7 3, 4 dichloroaniline (distilled and pure, melting point $71^\circ\text{--}72^\circ\text{C}$), (see 7.1).

4.2.8 Antifoam agent (polysiloxane derivative).

4.2.9 Perchloroethylene (tetrachloroethylene).

4.2.10 Distilled water.

5. PROCEDURE

5.1 Preparation of standard reference solution.

5.1.1 Dichloroaniline-hydrochloride stock solution. On an analytical balance, weigh 162.0 ± 0.1 milligrams (mg) of 3, 4 dichloroaniline. Place in a 1000 cc volumetric flask. Add 10 cc of 37.5 percent concentrated hydrochloric acid. Heat the flask in a water bath with boiling water and keep shaking the flask until the dichloroaniline is completely dissolved. Dilute to volume with distilled water 18° to 27°C .

5.1.2 Standard reference solution. Pipette 20 cc of dichloroaniline-hydrochloride stock solution (5.1.1) into a 1000 cc volumetric flask. Add 30 cc of distilled water (7° to 10°C). Cool the solution to 7° to 10°C , add 8 cc 1.0 N hydrochloric acid and 2 to 3 cc of 0.1 N sodium nitrite. Test with Congo Red test paper and the paper should turn dark blue instantly. Also test with Potassium Iodide-Starch test paper and the paper should turn black instantly. Agitate the solution thoroughly and keep at 7° to 10°C for exactly 20 minutes out of direct light. Add 30 cc of 1.0 N sodium bicarbonate solution to effect neutrality while maintaining the cold temperature. Check for neutrality with both red and blue litmus paper. Immediately add 3 cc of 0.01 M benzoyl-H-acid and agitate for two minutes to effect good coupling. Dilute to volume with distilled water. Determine absorbence of this standard reference solution using a filter photometer or spectrophotometer. Maximum absorbence occurs at approximately 505 nanometers. When using a filter photometer, a green filter having a maximum transmission at approximately 500 nanometers should be used.

5.2 Weight of dry specimen. The specimen shall be placed in a weighing bottle, dried in a circulating air oven at a temperature of 105° to 110°C cooled in a desiccator, and weighed. Repeat this cycle until a weight is obtained that is constant to ± 0.001 gram. This is the "weight of the dry specimen" and in the calculation of results is indicated as "O".

5.3 Testing of specimens containing 100 percent wool.

5.3.1 Cut specimen into small pieces and place in a 250 cc round bottom distillation flask with a few glass beads. Add 130 cc of 2.0 N potassium hydroxide and two drops of antifoam agent. Grease all ground glass connecting joints of distilling apparatus with silicone grease. Assemble the complete distilling apparatus as shown in Figure 2015 so that the distillate is collected through a funnel into a 500 cc volumetric flask. Heat the distilling flask gently, using an electric heater with variable control, until the liquid boils, and boil gently for 10 minutes. Increase the heating until vapor passes through 250 cc trap bulb and connecting arm and into the condenser. Continue distillation until approximately 100 cc of distillate has been collected. The distillate must not be contaminated by any carry-over of the liquid being distilled. Cool distillate to 7° to 10°C. Add 8 cc of 1.0 N hydrochloric acid and 2-3 cc of 0.1 N sodium nitrite. Agitate and test with Congo Red test paper; paper turns dark blue instantly. Test with Potassium Iodide-Starch test paper; paper turns black instantly. Agitate solution thoroughly and keep at 7° to 10°C for 20 minutes while protecting from direct light. Add 30 cc of 1.0 N sodium bicarbonate solution and check solution for neutrality with red and blue litmus paper. Add 3 cc of 0.01 M benzoyl-H-acid and agitate 2 minutes to effect good coupling. Dilute to volume with distilled water and measure the absorbence using a filter photometer or spectrophotometer. Maximum absorbence occurs at approximately 505 nanometers. When using a filter photometer, a green filter having a maximum transmission at approximately 500 nanometers should be used.

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5.4 Testing specimens of polyester/wool blend.

5.4.1 Place the specimen in a 250 cc round bottom distillation flask and a few glass beads. Add 150 cc of perchloroethylene, boiling point 119° to 121°C and connect flask to a reflux condenser. Reflux at the boil for two hours. Remove the specimen and rinse for one minute in 100 cc

of clean perchloroethylene at approximately 100°C. Air dry the specimen. Cut specimen into small pieces and place in distilling flask and continue with the procedure described in 5.3.1.

5.5 Calculations.

5.5.1 Specimens containing 100 percent wool. The percent mothproofing agent on the wool fiber shall be calculated from the absorbance measurements as follows:

$$\text{percent mothproofing agent} = \frac{544 \times A_t}{A_s \times O \times P}$$

Where: A_s = absorbance of standard reference solution. (see 5.5.3)
 A_t = absorbance of test solution.
 O = original dry weight of specimen in mg (5.2).
 P = Proportion of wool in the sample, expressed as a decimal to the nearest 0.01

5.5.2 Specimens containing polyester/wool blend. The percent mothproofing agent on the wool fiber shall be calculated from absorbance measurements as follows:

$$\text{percent mothproofing agent} = \frac{544 \times A_t \times 1.06}{A_s \times O \times P}$$

Where: A_s = absorbance of standard reference solution. (see 5.5.3)
 A_t = absorbance of test solution.
 O = original dry weight of specimen in mg (5.2).
 P = proportion of wool in the sample, expressed as a decimal to the nearest 0.01.

5.5.3 Where spectrometric measurements are taken from a transmission scale the absorbance shall be calculated as:

$$A = \log_{10} 1/T.$$

Where: T = transmission measurement.

A = Absorbance.

6. REPORT

6.1 The percent mothproofing agent content of a sample unit shall be reported as the average of values for the specimens tested from the sample unit.

6.1.1 The individual values for each individual specimen used to calculate the average shall also be reported.

7. NOTES

7.1 Materials required for testing. The 3, 4 dichloroaniline, the benzoyl-H-acid, and the 250 cc trap bulb and connecting arm may be obtained from Geigy Dyestuffs, Ardsley, New York 10502.

7.2 This method determines the content of a 100 percent active (pure) material.

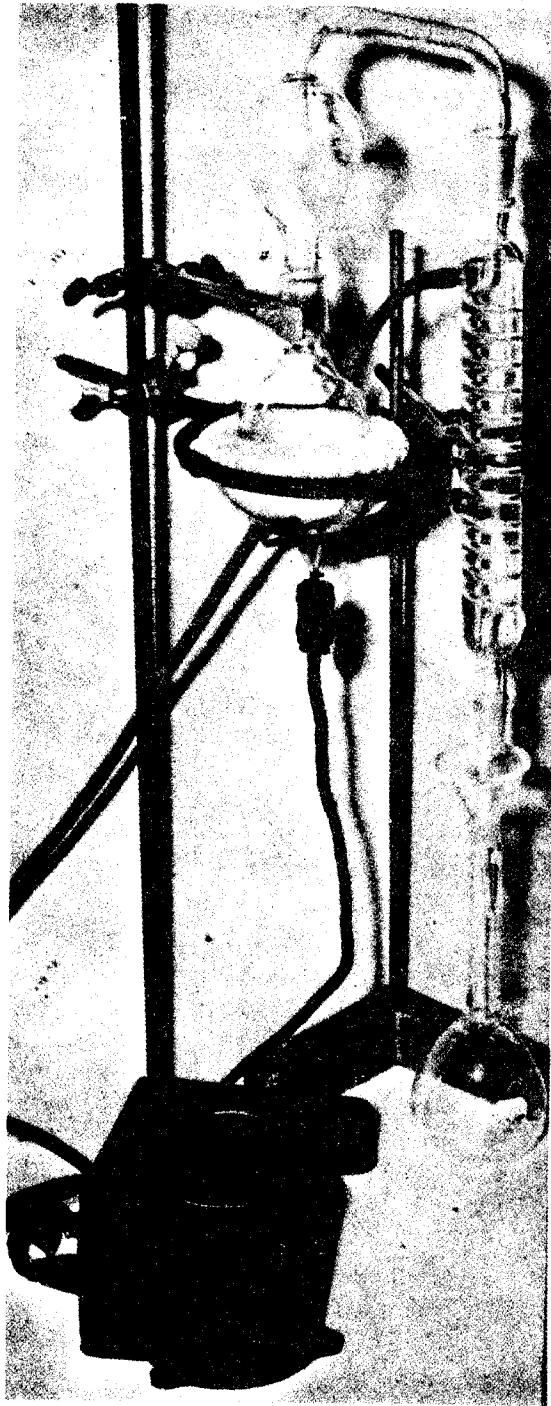


FIGURE 2015 - Distilling apparatus.

BECKER VALUE OF CORDAGE FIBER

1. SCOPE

1.1 This method is intended for evaluating the reflectance of abaca' (Manila "Hemp") fiber. It is applicable to raw fiber and to fiber from cordage.

2. TEST SPECIMEN

2.1 The test specimen shall be 24 grams of a composite sample. When cordage is to be tested, the composite sample is prepared from 3 pieces, 12 inches (30.5 cm) long taken from a sample unit not less than 2 feet (61 cm) from each other. If rope, yarn or fiber is to be tested, take enough 12 inch (30.5 cm) lengths of the yarn or fiber, well distributed over the sample unit, to give a bundle 1 inch (25.4 mm) in diameter.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in this material specification, four specimens shall be tested from each sample unit.

4. APPARATUS AND REAGENTS

4.1 Reflectometer. A reflectometer shall be used whose geometrical and spectral characteristics conform to requirements for the measurement of Becker Value (see 7.4).

4.2 Standards. Reflectance standards of porcelain enamel which have been calibrated relative to magnesium oxide on a reflectometer known to conform to the geometric and spectral requirements of the definition for Becker Value (see 7.1) shall be used, (see 7.2). For accurate results, a reference standard must have the same spectral character and approximately the same Becker Value as that of the fiber being tested (see 7.3).

4.3 Cuvette. A specimenholder at least 5/8 inch (15.9 mm) deep with clear glass or optical grade plastic window in the bottom large enough to receive the entire beam of the reflectometer. See Appendix II.

4.4 Glass or plastic sheet shall have the same thickness and spectral transmission as the bottom of the cuvette (see 4.3) and having the length and width of the reflectance standard (see 4.2) preferably cut from the same piece as the bottom window of the cuvette for calibrating equipment. The glass or plastic sheet is inserted between the standard and receptor in reflectometers sensitive to geometric and optical changes caused by its emission.

4.5 Soxhlet extraction apparatus.

4.6 Reagent.

4.6.1 Petroleum ether, technical.

5. PROCEDURE

5.1 Preparation of specimen.

5.1.1 Cut 3 pieces from a sample unit of rope to be tested, 12 inches (30.5 cm) long. Open up a piece of rope and remove the paper marker if present. Untwist the strands and the yarns and make a cylindrical bundle including all the fibers in the cross-section of the rope. Compress the bundle as much as possible. It may be helpful to wrap the bundle in heavy Kraft paper. The fibers must be cut to a length of $2 \text{ mm} \pm 0.5 \text{ mm}$ by slicing the bundle perpendicular to its long axis. Take care to avoid overheating and discoloration by too rapid cutting or a dull knife. Remove particles of paper, if used, from the cut fiber. Repeat this procedure for the 3 pieces cut from the sample unit.

5.1.2 Combine 8 gram portions of the cut fibers from each of the 3 pieces of rope and mix thoroughly.

5.1.3 Place approximately 10 grams of this composite sample in the thimble of the Soxhlet apparatus and extract by refluxing with petroleum ether for 2 hours. The solvent should be recycled approximately 15 times per hour.

5.1.4 Spread the extracted fibers out on a clean sheet of quantitative filter paper and allow them to dry completely at room temperature, preferably overnight.

5.2 Forming specimen in cuvette.

5.2.1 Carefully sprinkle the prepared specimen evenly into the cuvette from a spatula or folded sheet of paper. Do not handle the fibers with the fingers. When the window in the cuvette is evenly covered to a depth of about 1/16 inch (1.6 mm) add additional fibers more rapidly to a depth of 1/2 inch (12.7 mm). Do not tamp or pack the fibers. The fiber surface against the window of the cuvette constitutes the specimen measurement.

5.3 Becker Value.

5.3.1 Turn on the reflectometer and allow the equipment to warm up for about 1/2 hour to bring the response to a steady rate.

5.3.2 Calibrate the reflectometer as follows: Cover the reflectance standard, (see 4.2), with the glass or plastic sheet where necessary, (see 4.4), and place the combination over the specimen aperture of the reflectometer. Read the instrument. If the indicated reflectance differs from that of the reflectance standard, adjust the apparatus to give the correct reading.

5.3.3 If a Gardner Photometric Unit is being used, place the back (fig. 3810B) on the filled cuvette and place the cuvette with the window on the center of the specimen aperture of the reflectometer; completely covering it. Read the reflectance. Rotate the cuvette through 90 degrees and read the reflectance again. A large difference noted in rotating the cuvette indicates that the fibers are not arranged at random and the cuvette should be emptied and refilled. If the two readings agree within 0.3 Becker Value, check the calibration as in 5.3.2. If the calibration has not changed more than 0.3 Becker Value during the measurements, the two readings are acceptable. Average them to obtain a single value for the specimen. If the calibration has changed more than 0.3 Becker Value, the reflectance measurements must be repeated.

5.3.4 If a Photovolt Reflection Meter is being used, place the filled cuvette carefully on the center of the specimen aperture completely covering it, and take a reading. Raise the cuvette 1/4 inch (6.35 mm) above the surface of the reflectometer and allow it to drop back into place to settle the fiber. Take a second reading. Repeat this procedure until successive readings agree. Check the calibration with the reflectance standard as before and if it has not changed more than 0.3 Becker Value during the measurements, take the last reflectance reading to be the value for the specimen. Repeat the measurements if the calibration has changed more than 0.3 Becker Value.

6. REPORT

6.1 The Becker Value for the sample unit is the average of the values for the specimens measured.

6.2 The individual values used to arrive at the average value must be reported.

6.3 Report the Becker Value to the nearest whole number.

7. NOTES

7.1 Becker Value. The reflectance of a specimen relative to magnesium oxide when illuminated at 45° by CIE Source A passing through a Wratten 75 filter and viewed perpendicularly by a receptor whose spectral response is equivalent to that of the CIE Standard observer.

7.2 Reference standards may be obtained from the National Bureau of Standards, Washington, D. C. 20234 and Photovolt Corporation, 1115 Broadway, New York, N. Y. 10010.

7.3 A likely source of error in Becker Value measurements may be the departure of the spectral response of the reflectometer from the requirements of this method. A practical test for the conformance to spectral requirements may be made by adjusting the instrument to read correctly the Becker Value of a white reference standard then reading the value of a tan-colored standard that has nearly the same value as the fiber to be measured. The reading for the latter standard should be within a few tenths of the assigned values.

7.4 Gardner Photometric Unit when equipped with 45° degree exposure head and with Corning-403 and Wratten-75 filters between photocell and specimen is suitable. It may be obtained from Gardner Laboratories, Inc. Bethesda, Maryland 20014. The Photovolt Reflection Meter, when equipped with Corning 4010 and Corning 4308 filters, is suitable. It may be obtained from the Photovolt Corporation, 1115 Broadway, New York, N. Y. 10010. The Martens (visual) photometer with Wratten 75 filter over the eyepiece and Standard Light Source A of the CIE may be used as in the past, but measurement with this equipment is less precise and more time consuming than with the photo-electric equipment. Any photometer conforming to the general requirements may be used provided it is shown to yield Becker Values in close agreement with the values obtained with the equipment referred to above. (Appendix I).

APPENDIX 1

PHOTOMETRIC EQUIPMENT

To qualify photometric equipment for the Becker Value test, test at least 25 fiber specimens representing a range of Becker Values from 35 to 55 with it and with one of the photometers referred to in Section 7.4, of the test method without disturbing the surface measured. Plot the results as shown in Figure 3810A and fit a straight line to the data using the method of least squares. In the Becker Value range from 35 to 55, the discrepancy between this least squares line and a unit slope line through the zero intercept should not exceed 0.5 Becker Value.

Suggestions for the operation of photoelectric reflectometers follow. Locate the instrument on a bench or table free from vibration. Turn on the current at least $\frac{1}{2}$ hour before measurement to allow the instrument to warm up, with the resulting steadier readings and less frequent calibration. If the current is not steady the reflectometer should be operated with a special voltage regulator or from a storage battery. Check the zero setting before each use and reset if necessary. Cover the sample aperture completely so that no outside light can reach the photocell. Calibrate with the reflectance standard before and after measurements. Frequent rechecking of calibration is essential. Clean the optical system from time to time to remove dust and dirt and clean the filter before each use. If the component parts of the filter separate they should be recemented. Follow the instructions of the manufacturer of the reflectometer.

APPENDIX II

CUVETTE OR SPECIMEN HOLDER

A suitable cuvette for use with the Gardner Photometric Unit is shown in Figure 3810B. The surface measured is that of the fiber against the window in the bottom of the cell shown in cross-section in the upper part of "a". The fiber is held against the window by the back of the cell shown in "b". A cell for use with the Photovolt Reflection Meter may be made from a one-inch length of 35 millimeter (outside diameter) Pyrex glass tubing, 2 millimeters wall thickness. The window is made from ophthalmic crown glass, Code #8361 (Corning) or equivalent, 1.6 millimeters plus or minus 0.1 millimeter thick cemented to one end of the Pyrex tube. Clean cuvettes periodically with a detergent and water followed by rinsing with alcohol and ether and drying in a stream of air. After each use, blow out with air to remove all fibers and dust. Do not wipe with cloth or soft paper as this will generate static electrical charges, and result in non-random distribution of cut fibers, and cause incorrect reflectance values.

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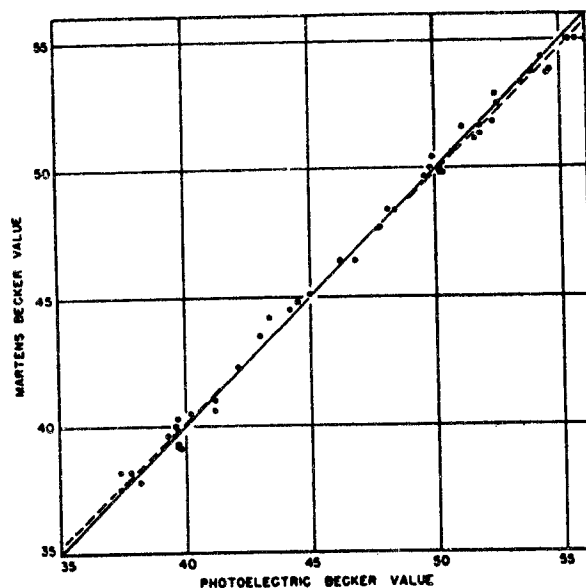
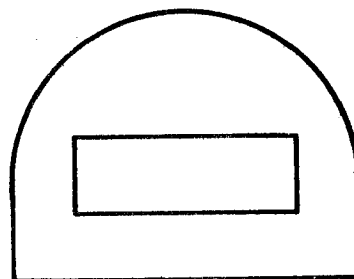
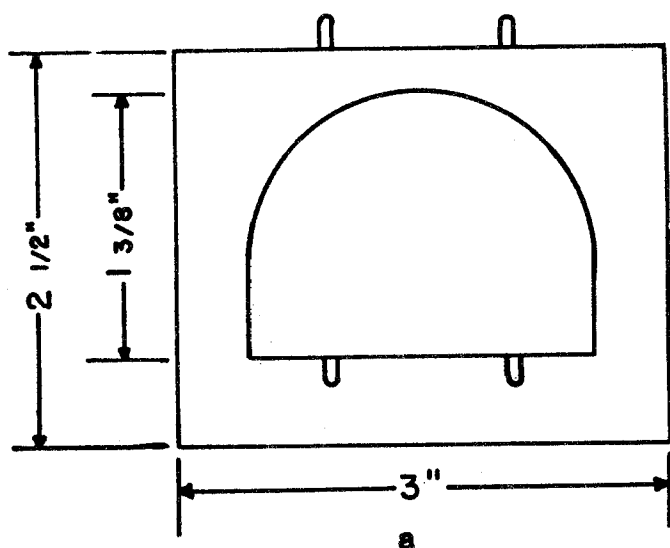
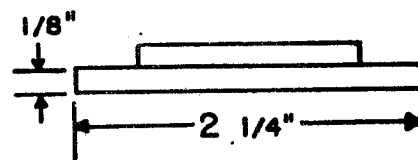


FIGURE 3810A - Photoelectric Becker values plotted against visual values.

Broken line represents the least-square line.



b

- a. Body of cell.
- b. Back of cell.

FIGURE 3810B - Cuvette used with Gardner photometric unit.

The back is held in place by the tension of rubber bands which are looped over the protruding studs in "a" and contact back on the rectangular raised portion.

LENGTH-WEIGHT RELATION; THREAD; YARDS PER POUND

1. SCOPE

1.1 This method is intended for determining the length per pound (length per kilogram) of sewing thread taken from such packages as spools or cones.

2. TEST SPECIMEN

2.1 The specimen shall be a skein of thread. Unless otherwise specified in the material specification, each skein of heavy thread shall contain not less than 30 yards (27.4 m); each skein of machine or basting thread shall contain not less than 120 yards (109.7 m).

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specifications, five specimens shall be tested from each sample unit.

4. APPARATUS

4.1 Reel, accurate to 0.1 percent, equipped with means for recording length, applying tension, and spreading the thread evenly on the reel.

4.2 Analytical balance or a grain-yarn scale, accurate to +0.25 percent.

5. PROCEDURE

5.1 Unless otherwise specified, the specimens tested shall be conditioned and tested under Standard Conditions in accordance with Section 4 of this Standard.

5.2 For threads wound on spools, cones, or similar put-up, the thread shall be drawn from the top of the package at a speed of 100 to 300 revolutions per minute of the reel. The thread shall be passed through the guides in such a way that the tension in the running yarn is sufficient to straighten it, but not high enough to cause serious stretching. If the reel has only one pig-tail guide per skein, tension shall be applied by taking one full wrap around the guide. If the reel has two or more guides, the thread shall pass straight through the guides onto the reel, the angle of the guides supplying necessary tension.

5.3 For threads on parallel tubes and large flanged spools, large tubes, certain warp wound bobbins or similar put up, the thread shall be drawn from the side at a speed of 20 to 30 revolutions per minute of the reel. Judgement must be used in applying tension on threads having a small or large amount of twist.

5.4 The finishing end of the skein shall be tied to the starting end of the skein in such a manner that the knot will not add additional length to the reel skein.

5.5 The prepared skein shall be weighed, using an analytical balance or a grain-yarn scale, to an accuracy of ± 0.25 percent of the total weight.

5.6 Calculations. The yards per pound (meters per kilogram) shall be calculated as follows:

$$\text{Yards per pound} = \frac{7000 \times \text{number of yards in specimen}}{\text{Weight of specimen in grains}}$$

OR

$$\text{Yards per pound} = \frac{453.6 \times \text{number of yards in specimen}}{\text{Weight of specimen in grams}}$$

OR

$$\text{Meters per kilogram} = \frac{\text{Number of meters in specimen}}{\text{Weight of specimen in kilograms}}$$

6. REPORT

6.1 The yards per pound (meters per kilogram) of a sample unit shall be the average of the specimens tested from the sample unit and shall be reported to the nearest yard (meter).

6.2 The individual values used to arrive at the average shall also be reported.

YARN NUMBER (LINEAR DENSITY) OF YARN FROM PACKAGE

1. SCOPE

1.1 This method is intended for determining the yarn number (linear density) of yarn taken from such packages as cones, cops, bobbins, tubes, and similar put-up. It is applicable to single and plied yarns.

2. TEST SPECIMEN

2.1 The test specimen shall be a skein of yarn. The specimen size shall be as follows:

2.1.1 Indirect system of units (length-weight ratio).

2.1.1.1 Skein length of all singles yarns shall be 120 yards, (109.7 meters).

2.1.1.2 Skein length of plied yarns shall be as follows:

Plied Yarn Equivalent Singles Numbers

Cotton and Spun Rayon	Linen and Wool Cut	Worsted	Wool Run	Length to Reel for Test	
				Yards	Meters
20 and above	56 and above	30 and above	11 and above	60	55
3 to 20	8.4 to 56	4.5 to 30	1.6 to 11	24	22
Below 3	Below 8.4	Below 4.5	Below 1.6	12	11

Direct System of Units (Weight per Unit Length) Filament Yarns

Below 130 denier	300	275
130 to 650 denier	120	110
650 denier and above	15	14

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, four specimens shall be tested from each sample unit.

4. APPARATUS AND METHOD CITED

4.1 Apparatus.

4.1.1 Yarn reel accurate to ± 0.1 percent, equipped with means to record length, applying tension, and spreading the yarn evenly on the reel.

4.1.2 Analytical balance or grain-yarn scale, accurate to ± 0.25 percent.

4.2 Method cited.

4.2.1 Method 4054, Twist and Twist Contration; Ply Yarns.

5. PROCEDURE

5.1 Unless otherwise specified, the specimens tested shall be conditioned and tested under Standard Conditions in accordance with Section 4 of this Standard.

5.2 For yarn wound on cones, filling wound bobbins and cops, and small flange spools or tubes, the yarn shall be drawn from the top of the package at a speed of 100 to 300 revolutions per minute of the reel. The yarn shall be passed through the guides in such a way that the tension in the running yarn is sufficient to straighten it, but not high enough to cause serious stretching. If the reel has only one pigtail guide per skein, tension shall be applied by taking one full wrap around the guide. If the reel has two or more guides, the yarn shall pass straight through the guides onto the reel, the angle of the guides supplying the necessary tension.

5.3 For packages such as large flanged spools, large tubes, certain warp wound bobbins or similar put-up, the yarn shall be drawn from the side at a speed of 20 to 30 revolutions per minute of the reel. If the reel has two or more guides, the yarn shall pass straight through the guides onto the reel. Judgement must be used in applying tension on yarns having a small or large amount of twist.

5.4 The finishing end of the skein shall be tied to the starting end of the skein in such a manner that the knot will not add additional length to the reeled skein.

5.5 The prepared skein shall be weighed using an analytical balance or grain-yarn scale to an accuracy of ± 0.25 percent of the total weight.

5.6 Calculations. The yarn number, using the weight and length of the skein, shall be calculated using the following formulas:

Indirect system:

$$\text{Yarn Number } N = \frac{L}{W} \times \frac{453.6}{Y} \quad (\text{or}) \quad N = \frac{L}{W_1} \times \frac{7000}{Y}$$

$$\text{Yarn Number } N_1 = \frac{L \times P}{(1-C/100)} \times \frac{453.6}{W \times Y} \quad (\text{or}) \quad N_1 = \frac{L \times P}{(1-C/100)} \times \frac{7000}{W_1 \times Y}$$

Direct system - Denier:

$$\text{Denier } N = \frac{W \times 9840}{L} \quad (\text{or}) \quad \frac{W \times 9000}{L_1}$$

$$\text{Denier } N_1 = \frac{W \times 9840}{(1-C/100) L} \quad (\text{or}) \quad \frac{W \times 9000}{(1-C/100) L_1}$$

$$\begin{aligned} \text{Tex Units} &= \frac{310.034}{\text{woolen run number}} \\ &= \frac{590.541}{\text{cotton hank number}} \\ &= \frac{885.812}{\text{worsted hank number}} \\ &= \frac{1653.5}{\text{linen lea number}} \\ &= \frac{1653.5}{\text{wool cut number}} \\ &= \frac{\text{denier}}{9} \quad (\text{synthetics}) \end{aligned}$$

Where:

- N - equivalent single yarn number of plied yarn or number of single yarn.
N₁ - true single yarn number, i.e., single yarn number prior to plying.
L - length of single or ply yarn in yards.
L₁ - length of single or ply yarn in meters.
W - weight of skein in grams.
W₁ - weight of skein in grains.

453.6 grams in one pound.
7000 grains in one pound.

- P - number of plies in yarn.
C - twist contraction in plying, percent (see Method 4054).
Y - yards of No. 1 yarn in one pound. The value of Y for indirect system is:

840 for cotton system (cotton, spun rayon)
300 for linen and wool, "cut" system
560 for worsted
1600 for wool, "run" system

Constant for direct system:

9000 for denier, grams per 9000 meters
9840 for denier, grams per 9840 yards

5.6.1 The yarn number of a specimen shall be based on its equivalent single yarn number. If the finished yarn is made up of plied components, the yarn number is expressed as a multiple of the single yarn number for the component yarns.

Examples:

Cotton, spun rayon and blends - A single yarn with an equivalent yarn number of 60 would be expressed as 60/1. A plied yarn composed of 3 single yarns of No. 60 and whose equivalent single yarn number may be for example, 19 or 20 shall be expressed as 60/3.

Wool cut or run, worsted and linen - A single yarn with an equivalent yarn number of 60 would be expressed as 1/60. A plied yarn composed

of 3 single yarns of No. 60 and whose equivalent single yarn number may be, for example, 19 or 20 shall be expressed as 3/60.

Filament yarn - A plied yarn of 4 single yarns of 210 denier, 3⁴ filament shall be expressed as 210/3⁴/4 ply.

6. REPORT

6.1 The yarn number of sample unit shall be the average of the specimens tested.

6.1.1 The yarn number in the indirect system shall be reported to the nearest 0.1 number for yarn numbers 0 to 12 inclusive, and to the nearest whole number for yarn numbers above 12.

6.1.2 Yarn number in the direct system shall be reported to the nearest whole number.

6.2 The individual values used to arrive at the results shall also be reported.

7. NOTES

7.1 Caution should be used when making plied yarn determinations because the amount of contraction due to twist will have an effect on the yarn number.

DIRECTION OF TWIST: YARN, THREAD, CORDAGE

1. SCOPE

1.1 This method is intended for determining the direction of twist of yarn, thread, and cordage.

2. TEST SPECIMEN

2.1 Unless otherwise specified in the material specification, the specimen shall be any convenient length.

3. NUMBER OF DETERMINATIONS

3.1 Unless otherwise specified in the material specification, three specimens shall be tested from each sample unit.

4. APPARATUS

4.1 No special apparatus is required for this method, but a magnifying glass and pick needle may be necessary for testing extremely fine yarns.

5. PROCEDURE

5.1 The specimen shall be held in a vertical position and the direction of the slope of the twist spirals observed.

6. REPORT

6.1 Direction of twist of specimen. The direction of the twist of each specimen shall be expressed as "S" twist if the spirals conform in direction of slope to the central portion of the letter "S", and "Z" twist if the spirals conform in direction of slope to the central portion of the letter "Z" (see Figure 4050).